

## PATENT SPECIFICATION



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" " Oct. 4, 1920. No. 28,102/20.

One Complete Specification Left: Apr. 12, 1921.

Complete Accepted: Dec. 12, 1921.

### PROVISIONAL SPECIFICATION.

No. 15,974, A.D. 1920.

### Improvements in and relating to the Employment of Antiseptic, Disinfectant, or Preservative Agents.

We, ROBERT LAUDER MACKENZIE WALLIS, of 55, Townshend Road, St. John's Wood, London, N.W. 8, a subject of the King of Great Britain and Ireland, and ATMOSTEROL LIMITED, of The Moorings, Leigh-on-Sea, in the County of Essex, a company registered under the laws of Great Britain, do hereby declare the nature of this invention to be as follows:—

This invention relates to the employment of antiseptic, disinfectant, or preservative agents and particularly to those which are normally solid.

Experience has shown that considerable difficulties are experienced in securing the effective distribution of disinfectant, or preservative agents which are normally solid and which are insoluble in the fluid portions of the bodies or materials in the treatment of which they are employed.

This is particularly the case with volatile organic, antiseptic agents or disinfectants which are normally crystalline solids and more especially with those which volatilize at temperatures below their melting points, as for instance, thymol.

In employing such antiseptic agents and disinfectants it is wholly impracticable in many cases to soak the material to be treated in a solution or emulsion of the antiseptic agent or disinfectant and consequently attempts have been made to apply them, by subjecting the material to be treated to the action of such agents in the form of vapour or in an atomized state. These methods of using such

agents are however not wholly satisfactory as the vapour condenses or the particles of atomized material coalesce to form relatively large drops or crystalline particles with the result that the material rapidly condenses and collects on the floors or walls of the chamber or vessel in which the treatment is carried out, and therefore does not come into contact with the whole of the material it is desired to treat, or it condenses or collects on the material in relatively large particles with the result that while some portions of the surface of the material are treated with the antiseptic or disinfectant agent, there are relatively large areas which are not so treated intervening between such portions.

According to the present invention the antiseptic or disinfectant agents are employed in association with conveyers or distributing agents.

Thus, in accordance with the invention an antiseptic or disinfectant agent which is to be employed in the form of vapour or in the atomized state is employed in association with a body adapted to act as a conveyor or distributing agent, in that it restrains the drops or particles of the agent from coalescing and assists in uniformly distributing the agent over the surface of the material to be treated.

The materials employed as conveyers or distributing agents preferably possess in a relatively small degree the capacity to coagulate or harden albumens but not definitely to effect the precipitation of such bodies.

In carrying the present invention into effect an alcohol, aldehyde, ketone or allied body having a relatively high molecular weight may be employed.

5 We may, for instance, employ an aliphatic alcohol of relatively high molecular weight as, for instance, butyl alcohol which acts as a solvent for thymol, carvacrol and other bodies  
10 allied with the terpenes or which may be derived from vegetable sources or the homologues or isomers of such bodies, synthetically produced or not.

15 Butyl alcohol, it has been found, not merely acts as a solvent for such bodies but also enables them to be emulsified with water and the alcohol further does not possess that property of coagulating protein or other bodies contained in the  
20 blood of animals as is possessed by the lower alcohols, for instance, ethyl alcohol and the lower aldehydes. Butyl alcohol has been found to aid considerably in

forming a mist or fog when, for instance, a solution of thymol or the like in butyl 25 alcohol is vaporised or atomized into a chamber and therefore it enables the material contained therein to be more thoroughly subjected to the action of the antiseptic agent or disinfectant than 30 when the use of a carrier or distributing agent is dispensed with.

While the employment of butyl alcohol has been referred to above in detail, it is to be understood that the 35 invention is not limited to the employment of this body which is referred to merely by way of example for the purpose of indicating the properties possessed by the preferred conveyers or distrib- 40 ing agents employed in accordance with the invention.

Dated this 12th day of June, 1920.

MARKS & CLERK.

#### PROVISIONAL SPECIFICATION.

No. 28,102, A.D. 1920.

#### Improvements in and relating to the Production of Organic Compounds Adapted for use as Antiseptics and to Processes of Sterilisation, Disinfection, and the like.

45 We, ROBERT LAUDER MACKENZIE WALLIS, of 55, Townshend Road, St. John's Wood, London, N.W. 8, a subject of the King of Great Britain and Ireland, and ATMOSTEROL LIMITED, of  
50 The Moorings, Nelson Drive, Leigh-on-Sea, in the County of Essex, a company registered under the laws of Great Britain, do hereby declare the nature of this invention to be as follows:—

55 This invention relates to the production of organic compounds adapted for use as antiseptics, and to processes of sterilisation, disinfection, or antiseptics, and particularly to preserving meat and other  
60 foods.

The invention is based upon the observation that the higher phenols, such as thymol, carvacrol and the like, and alcoholic aldehydic and etheric bodies of the aromatic or terpene groups such as  
65 are present in essential oils yield with the higher aliphatic alcohols containing for instance, three to six carbon atoms under the action of oxygen, bodies or compositions possessing powerful antiseptic prop-  
70 erties.

The invention may be explained by reference to thymol.

This compound we have found yields with higher alcohols under the action of 75 oxygen or other oxidising agents bodies which are apparently compounds of obscure chemical composition which possess powerful antiseptic properties.

When, for instance, a mixture of butyl 80 alcohol and thymol in certain proportions as, for instance, 100 ccs. butyl alcohol and 100 gms. thymol, is allowed to stand in contact with air, a reddish oily liquid is formed, apparently in consequence of 85 certain changes produced by oxidation.

An oily liquid of a similar character may be produced in accordance with the invention by adding to a mixture of thymol and butyl alcohol an oxidising 90 agent, such as hydrogen peroxide or ozonic ether.

By the employment of other higher alcohols as, for instance, propyl and amyl alcohol instead of butyl alcohol, similar 95 results may be obtained.

These oily liquids mix with water to form emulsions. They are themselves

powerful antiseptics as also are their emulsions with water.

The emulsions may be diluted in accordance with the invention and protective colloids or emulsifying agents, such as soap, may if desired be incorporated therewith.

As evidence in support of the view that new compounds are formed in the conditions referred to, the following facts may be adduced:—

(1) Change of colour;

(2) Change of odour, the reddish oil being substantially odourless;

(3) On heating the odour returns and the mixture becomes colourless;

(4) On distillation a liquid product is obtained which on standing develops heat, solid thymol separating out.

Meat may be preserved in accordance with the invention with the aid of this and like bodies and it is found that the presence of oxygen may be desirable when these bodies are employed for this purpose.

When, for instance, the meat is packed, hung or otherwise stored in a chamber, additional oxygen may be supplied to the atmosphere in the chamber.

The introduction of such additional oxygen may be effected intermittently or continuously so as to replace that which may be absorbed or consumed by the material stored or so as to maintain in the chamber an atmosphere containing a larger proportion of oxygen than is present in air.

The oily compound obtained in accordance with the invention may be employed by vapourising or atomising the material or emulsions thereof without the application of heat and subjecting the meat to the action of the material in the vaporised or atomised state in conjunction with cold storage or not.

The material may be atomised by the employment of oxygen under pressure, as in this way the supply of oxygen to the surface of the meat may be conveniently effected.

The emulsions in accordance with the invention may be sprayed, brushed or otherwise applied to meat, or the meat may be dipped into the emulsions, or the latter may be added to the pickle or liquor employed for curing the meat.

The invention also comprises the use of the material for disinfecting houses, public carriages, places of entertainment, tube railways and other structures used by a number of people, by introducing the material in an atomised state either

continuously or intermittently by way of the ventilating system or otherwise.

The invention also extends to the employment of ice obtained by adding the material or an emulsion thereof to water and freezing the water in preserving fish or other material which may be packed in contact with ice.

The process which comprises bringing together higher phenols such as thymol, carvacrol and the like or alcoholic, aldehydic, and etheric bodies of the aromatic and terpene groups such as are present in essential oils and higher aliphatic alcohols containing, for instance, three to six carbon atoms in the presence of oxygen or other suitable oxidising agent may be applied in accordance with the invention to the production of such phenols or other bodies of the classes indicated in a pure state by first forming the oxygen compound and then decomposing it under the action of heat.

Thus, for instance, commercial thymol may be brought together with butyl alcohol in the presence of oxygen or a suitable oxidising agent, in the manner above indicated, and the oily product then subjected to distillation, preferably under reduced pressure. The first fraction of the distillate will consist mainly of butyl alcohol, after which a higher boiling fraction will be obtained from which, as above indicated, thymol will separate out in large clear crystals, the residue in the distillation vessel containing the impurities present in the crude thymol.

As will be understood, the above description is given for the purpose of explaining the nature of the invention and that therefore the detailed description with respect to the production of the antiseptics, their method of use and so forth are supplied for the purpose of indicating the nature of the invention and not with a view to indicating the limits of its scope.

It may here be pointed out that in the Specification of the prior Application for Letters Patent No. 15,974 of A.D. 1920, there is described a process of employing antiseptic, disinfectant or preservative agents including thymol in which, among other bodies, butyl alcohol is stated to be employed as a conveyor or distributing agent. From the work since carried out it would appear that the application of the term "conveyor or distributing agent" to the butyl alcohol or other bodies when so employed is inappropriate and that these bodies enter into chemical

4 combination with the thymol or other preservative agents and oxygen, yielding compounds such as are described herein, and that the better results which are  
 5 obtained in accordance with the process described in the aforesaid specification as compared with the results obtained when, for instance, thymol alone is used,

are to be attributed to the formation of such compounds as are produced by the action of oxygen on the mixture of thymol and butyl alcohol. 10

Dated this 4th day of October, 1920.

MARKS & CLERK.

X COMPLETE SPECIFICATION.

Improvements in and relating to the Production of Organic Compounds  
 Adapted for use as Antiseptics and to Processes of Sterilisation,  
 Disinfection and the like.

15 We, ROBERT LAUDER MACKENZIE WALLIS, of 55, Townshend Road, St. John's Wood, London, N.W. 8, a subject of the King of Great Britain and Ireland, and ATMOSTEROL LIMITED, of  
 20 The Moorings, Nelson Drive, Leigh-on-Sea, in the County of Essex, a company registered under the laws of Great Britain, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by  
 25 the following statement:—

This invention relates to the employment of antiseptic, disinfectant or preservative agents, and particularly to those which are normally solid.

30 Experience has shown that considerable difficulties arise in securing the effective distribution of disinfectant or preservative agents which are normally solid and which are insoluble in the fluid portions of the bodies or materials in the treatment of which they are employed.

This is particularly the case with volatile organic antiseptic agents or disinfectants which are normally crystalline solids and more especially with those which volatilise at temperatures below their melting points, as, for instance,  
 45 thymol.

According to the present invention the higher phenols such as thymol, carvacrol and the like and alcoholic, aldehydic and etheric bodies, of the aromatic and  
 50 terpene groups such as are present in essential oils, are employed in association with aliphatic monohydric alcohols having a relatively high molecular weight, for instance having from three to six carbon atoms or the corresponding aldehydes or ketones. We may, for instance  
 55 employ butyl alcohol, which acts as a solvent for thymol, carvacrol and like phenolic bodies, such as are present in essential oils.

Iso-butyl alcohol, it has been found, for instance, not merely acts as a solvent for such bodies but also enables them to be emulsified with water and the alcohol further does not possess the property of  
 65 coagulating proteins or other bodies contained in the blood of animals as is possessed by the lower alcohols, for instance ethyl alcohol and the lower aldehydes. Butyl alcohol has been found to  
 70 aid considerably in forming a mist or fog when, for instance, a solution of thymol or the like in iso-butyl alcohol is vaporised or atomised into a chamber and therefore it enables the material con-  
 75 tained therein to be more thoroughly subjected to the action of the antiseptic agent or disinfectant than when the use of a carrier or distributing agent is dispensed with.

The invention is further based upon the observation that the higher phenols, such as thymol, carvacrol and the like and alcoholic, aldehydic, etheric bodies of the aromatic and terpene groups such as  
 80 are present in essential oils yield with the aliphatic monohydric alcohols having a relatively high molecular weight, for instance having from three to six carbon atoms, under the action of oxygen and at  
 85 low or moderate temperatures, bodies or compositions possessing powerful antiseptic properties.

This phase of the invention may be explained by reference to thymol.

This compound we have found yields with higher alcohols, such as the propyl, butyl and amyl alcohols, under the action  
 95 of oxygen or other oxidising agents, bodies which are apparently compounds of obscure chemical composition and which possess powerful antiseptic properties.

When, for instance, a mixture of iso-butyl alcohol and thymol in certain proportions as, for instance, 100 ccs. iso-  
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butyl alcohol and 100 gms. thymol is allowed to stand in contact with air and at room temperature, a reddish oily liquid is formed, apparently in consequence of certain changes produced by oxidation.

An oily liquid of a similar character may be produced in accordance with the invention by adding to a mixture of thymol and butyl alcohol an oxidising agent, such as hydrogen peroxide or ozonic ether.

By the employment of other higher alcohols as, for instance, propyl and amyl alcohol instead of butyl alcohol, similar results may be obtained.

These oily liquids mix with water to form emulsions. They are themselves powerful antiseptics as also are their emulsions with water.

The emulsions may be diluted in accordance with the invention and protective colloids or emulsifying agents, such as soap, may if desired be incorporated therewith.

As evidence in support of the view that new compounds are formed in the conditions referred to, the following facts may be adduced:—

(1) Change of colour;  
(2) Change of odour, the reddish oil being substantially odourless;

(3) On heating the odour returns and the mixture becomes colourless;  
(4) On distillation a liquid product is obtained which on standing develops heat, solid thymol separating out.

Meat may be preserved in accordance with the invention with the aid of the thymol product above referred to and bodies similarly produced and which are of low toxicity or are non toxic, and it is found that the presence of oxygen may be desirable when these bodies are employed for this purpose.

When, for instance, the meat is packed, hung or otherwise stored in a chamber, additional oxygen may be supplied to the atmosphere in the chamber.

The introduction of such additional oxygen may be effected intermittently or continuously so as to replace that which may be absorbed or consumed by the material stored or so as to maintain in the chamber an atmosphere containing a larger proportion of oxygen than is present in air.

The oily compound obtained in accordance with the invention may be employed by vaporising or atomising the material or emulsions thereof without the application of heat and subjecting the meat to the action of the material in a finely

divided or atomised state in conjunction with cold storage or not.

The material may be atomised by the employment of oxygen under pressure, as in this way the supply of oxygen to the surface of the meat may be conveniently effected.

The emulsions in accordance with the invention may be sprayed, brushed or otherwise applied to meat, or the meat may be dipped into the emulsions or the latter may be added to the pickle or liquor employed for curing the meat.

The invention also comprises the use of the material for disinfecting houses, public carriages, places of entertainment, tube railways and other structures used by a number of people, by introducing the material in an atomised state either continuously or intermittently by way of the ventilating system or otherwise.

The invention also extends to the employment of ice obtained by adding the material or an emulsion thereof to water and freezing the water in preserving fish or other material which may be packed in contact with ice.

The process which comprises bringing together higher phenols such as thymol, carvacrol and the like and alcoholic aldehydic and etheric bodies of the aromatic or terpene groups such as are present in essential oils and an alcohol, having a relatively high molecular weight, for instance, having three to six carbon atoms, in the presence of oxygen or other suitable oxidising agent may be applied in accordance with the invention to the production of such bodies in a pure state by first forming the oxygen compound and then decomposing it under the action of heat.

Thus, for instance, commercial thymol may be brought together with iso-butyl alcohol in the presence of oxygen or a suitable oxidising agent in the manner above indicated and the oily product then subjected to distillation, preferably under reduced pressure, at for instance, temperatures between 50 and 60° C. under 100<sup>mm</sup> water column. The first fraction of the distillate will consist mainly of iso-butyl alcohol, after which a higher boiling fraction will be obtained from which, as above indicated, thymol will separate out in large clear crystals, the residue in the distillation vessel containing the impurities present in the crude thymol.

From the work we have carried out it would appear that the aliphatic alcohols of relatively high molecular weight and containing, for instance, from three to

six carbon atoms and the corresponding aldehydes and ketones referred to as conveyors or distributing agents enter into chemical combination with the thymol or other higher phenols and alcoholic, aldehydic and etheric bodies of the aromatic or terpene groups such as are present in essential oils, and oxygen, yielding compounds such as are described herein, and that the better results which are obtained in accordance with the process described in the Provisional Specification No. 15,974 dated June 12th, 1920, as compared with the results obtained when, for instance, thymol alone is used, are to be attributed to the formation of such compounds as are produced by the action of oxygen on the mixture of thymol and butyl alcohol.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:-

1. The improved method of using the higher phenols such as thymol, carvacrol and the like and alcoholic, aldehydic and etheric bodies of the aromatic or terpene groups such as are present in essential oils, as antiseptic, disinfectant and preservative agents in which the said bodies are employed in association with an aliphatic monohydric alcohol having a relatively high molecular weight, for instance, containing from three to six carbon atoms or the corresponding aldehydes or ketones.

2. The method of employing antiseptic, disinfectant and preservative agents as claimed in Claim 1, in which butyl alcohol is used in association with thymol, carvacrol and other higher phenols or alcoholic, aldehydic and etheric bodies of the aromatic or terpene groups such as are present in essential oils.

3. The process which comprises bringing together higher phenols such as thymol, carvacrol and the like and alcoholic, aldehydic and etheric bodies of the aromatic or terpene groups, such as are present in essential oils, and higher aliphatic alcohols as, for instance, iso-butyl alcohol, in the presence of oxygen or other suitable oxidising agent, and at relatively low temperatures.

4. A process as claimed in Claim 3, in which the compositions formed are decomposed under the action of heat in order to reproduce the higher phenols or alcoholic aldehydic or etheric bodies in a pure state, substantially as described.

5. Antiseptic, disinfectant and preservative compositions formed from the higher phenols, such as thymol, carvacrol and the like, and alcoholic, aldehydic and etheric bodies of the aromatic or terpene groups, such as are present in essential oils, and the higher alcohols under the action of an oxidising agent as claimed in Claim 3.

6. Antiseptic, disinfectant and preservative compositions as claimed in Claim 5, formed by the interaction of iso-butyl alcohol, a higher phenol and an oxidising agent as, for instance, oxygen, hydrogen peroxide or ozonic ether.

7. An antiseptic composition as claimed in Claim 6 formed from iso-butyl alcohol and thymol.

8. Antiseptic, disinfectant or preservative compositions comprising the bodies claimed in Claims 5 to 7 in the form of an emulsion.

9. The improved methods of disinfection and preservation, particularly of food-stuffs, substantially as hereinbefore described.

Dated this 12th day of April, 1921.

MARKS & CLERK:

SOURCE: (C) WPI / DERWENT

AN : 94-088588 [11]

MC : D03-A05

PN : JP6038678 A 940215 DW9411 A23B9/16 006pp

PR : JP920122950 920417

PA : (OKUB/) OKUBO T

DC : D13

IC : A23B9/16

TI : Rice preservative mfr. to avoid occurrence of mould, microorganisms etc. - by placing spice contg. at least allicin and capsaicin in a permeable vessel and placing with rice in a container.

AB : J06038678 In the method spice contg. at least allicin and capsaicin is put in a permeable vessel, and the vessel and rice are put together in a container such as rice chest or rice bag.

- USE - Mould microorganisms and noxious insects of stored grains can be prevented from occurring in rice, and oxidn. of rice is prevented.  
(Dwg.1/5)

